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## **Freshwater Reservoir Effect on Re-Dating of Eurasian Steppe Cultures: First Results for Eneolithic and Early Bronze Age North-East Kazakhstan**

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**Title:** Freshwater Reservoir Effect on Re-Dating of Eurasian Steppe Cultures: First Results for Eneolithic and Early Bronze Age North-East Kazakhstan

**Abstract:** Freshwater reservoir effects (FRE) can cause a major problem with radiocarbon dating human skeletal material in the Eurasian steppe. We present the first results of research into the extent of the FRE in the sites of Borly 4 (Eneolithic), and Shauke 1 and 8b (Early Bronze Age), North-Eastern Kazakhstan. AMS <sup>14</sup>C dating and stable isotope ( $\delta^{13}\text{C}$ ,  $\delta^{15}\text{N}$ ) analysis of associated groups of samples (32 samples, 11 groups in total) demonstrate that: a) the diet of the humans and fauna analysed was based on the C<sub>3</sub> foodchain with no evidence of a C<sub>4</sub> plant (such as millet) contribution; aquatic resources apparently were a continuous dietary feature for the humans; b) the first <sup>14</sup>C dates obtained for the Upper and Middle Irtysh River region attribute the Eneolithic period of the area to the 34<sup>th</sup>-30<sup>th</sup> c. BC, and the Early Bronze Age – to the 25<sup>th</sup>-20<sup>th</sup> c. BC; there is a ca. 450 years hiatus between the two periods; c) the maximum fish-herbivore freshwater reservoir offset observed equals 301±47 <sup>14</sup>C yrs. As such, <sup>14</sup>C dates from aquatic and human samples from the area need to be interpreted with caution as they are likely to be affected by the offset (i.e. appear older).

The paper also discusses the effect of a sodium hydroxide (NaOH) wash on  $\delta^{13}\text{C}$ ,  $\delta^{15}\text{N}$ , C:N<sub>atomic</sub> levels and collagen yields of the bone samples. Our results indicate a minor but significant effect of NaOH treatment only on C:N<sub>atomic</sub> ratios of the samples.

**Keywords:** Eurasian Steppe, NE Kazakhstan, Eneolithic, Bronze Age, Freshwater Reservoir Effects, sodium hydroxide (NaOH)

## Introduction

Archaeologists often sample human bone to radiocarbon (<sup>14</sup>C) date past societies especially when undertaking palaeodietary studies. However, in many cases a major source of uncertainty, the reservoir effect, hinders the accuracy of these dates. The effect occurs when a portion of the carbon in the diet comes from a non-atmospheric reservoir with a lower <sup>14</sup>C level (e.g. marine/freshwater). The difference between <sup>14</sup>C age of such a bone sample and that of a contemporaneous, purely terrestrial sample is termed the “reservoir offset”.

The extent of the marine reservoir effect is well-documented in terms of the average global marine reservoir correction (estimated as 400 years; Stuiver and Braziunas 1993), regional differences ( $\Delta R$ ) (compiled in the marine reservoir correction database; [www.calib.org/marine](http://www.calib.org/marine)), and its impact on human/animal bone samples through marine-based diet (Ascough et al. 2005; Shishlina et al. 2007, 2009, 2012, 2014). In contrast, research on the extent of the freshwater reservoir effect (FRE) is rather scarce. Most FRE studies have been focused on Europe and the European part of Russia (Cook et al. 2001, 2002; Fischer and

Heinemeier 2003; Olsen et al. 2010; Keaveney and Reimer 2012; Wood et al. 2013; Loughheed et al. 2013; Fernandes et al. 2014) and North America (Ingram and Southon 1996; Goodfriend and Flessa 1997; Culleton 2006), and only a few in Siberia and the Eurasian Steppe (Shishlina et al. 2007, 2009, 2012; Lillie et al. 2009; Higham et al. 2010; Nomokonova et al. 2013; Schulting et al. 2014). The main conclusions from this research are that FRE is highly variable geographically, spanning from zero to several thousand years, and that “each population thought to be affected by a FRE must be examined individually” (Wood et al. 2013, p. 163). However, exploring the phenomenon in particular areas, analyzing its extent or demonstrating its absence in non-affected regions can significantly improve chronological interpretations for these areas. The largest source of “old” carbon in freshwater is dissolved inorganic carbon from  $^{14}\text{C}$ -free carbonate minerals in the groundwater, as many sedimentary rocks are composed of skeletal fragments of marine organisms that died millions of years ago (Sveinbjörnsdóttir et al. 1995; Culleton 2006). The extent of the FRE in most regions is closely related to the geological composition of the underlying bedrock. High correlation between the FRE and carbonate alkalinity has been demonstrated for modern lakes in Britain and Ireland (Keaveney and Reimer 2012).

The main aim of this study is to improve the accuracy of  $^{14}\text{C}$  dating of archaeological (primarily Bronze Age) human bone by assessing the extent and diversity of the FRE in the key areas of Western and Southern Siberia and Kazakhstan. We also aim to explore palaeodietary and palaeoeconomic patterns of the populations, specifically the role of freshwater resources in human diet.

Siberia and the Eurasian steppe represent an unmatched economic, political and cultural interface between the East and West from early prehistory. The turbulent history of migrations and collisions of tribes and nations, rise of empires and fall of civilizations in the region has forged its unique heritage drawing enormous interest from the global academic community (Mair 2006; Anthony 2007; Kuzmina 2008; Beckwith 2009). A growing number of  $^{14}\text{C}$  ages from archaeological human bone samples appear considerably older than expected from traditional or established archaeological chronologies based on associated wood and/or charcoal  $^{14}\text{C}$  ages (Görsdorf et al. 2001; Alekseev et al. 2005; Hanks et al. 2007; Svyatko et al. 2009), possibly due to the FRE. Often particular human burials are of interest but they do not always have associated terrestrial material for dating. In many cases (e.g. plundered burials, graves without goods or wooden structures)  $^{14}\text{C}$  dating of human bone is the most feasible, and sometimes the only way to date particular sites. Furthermore, there is a large number of human bone  $^{14}\text{C}$  dates from past research, and it is important to assess their accuracy where a FRE correction is required. The refinement of radiocarbon dates through documenting the FRE in these regions is crucial for understanding the chronology of cultural transitions and development of these interactions. A number of recent studies (O'Connell et al. 2003; Privat et al. 2005; Shishlina et al. 2007; Svyatko et al. 2013; Murphy et al. 2013) have suggested a strong contribution of freshwater fish in the diet of prehistoric north Eurasian steppe populations, which complicates the interpretation of radiocarbon dates because of the possibility of a FRE.

### *Geographical Background*

The current stage of our research is focused on archaeological sites of North-East Kazakhstan, in particular – Pavlodar Oblast. Geographically, Pavlodar Oblast is a part of the Irtysh River Plain with Irtysh River being the main water artery. The 10-15 km wide river valley bears a large number of Irtysh tributaries and minor lakes and it is completely flooded during the spring high-water period. The area is also characterized by a large number of deflation basins filled with salt lakes. The bedrock of the region is mostly composed of alluvial

and lacustrine-alluvial Early to Middle Pleistocene deposits dominated by sandy kastanosems, with the well-defined carbonaceous layer at the depth of 30-50 cm (Esenov 1970). Modern alluvial deposits of various composition are soil-forming material for the Irtysh River flood plains; river terraces are widely composed of carbonaceous sandy kastanosems (Uryvaev 1959), while 30-50 % of the lake basin deposits represent complex alkaline sandy kastanosems (Esenov 1970).

Along the entire length of the Irtysh River, its water is carbonaceous and moderately hard (carbonate alkalinity generally varies within 100-160 mg/L throughout the year) and potable. The chemical composition of the river water is formed outside the Pavlodar Oblast, mainly in highlands of the Upper Irtysh Region (Uryvaev 1959); obviously, melted snow constitutes a proportion of the river water.

The area is mainly covered by dry steppes; the modern climate is continental. Summers are hot (mean  $T = 23^{\circ}\text{--}25^{\circ}\text{C}$ ;  $>40^{\circ}\text{C}$  max.), with shower rains; winters are cloudless 17-21 days/month with little snow (22 cm thick by February), mean winter temperature varies between  $-10^{\circ}\text{C}$  and  $-20^{\circ}\text{C}$  (min.  $-47^{\circ}\text{C}$ ; Esenov 1970). The mean annual precipitation averages around 240 mm (Uryvaev 1959). The area is dominated by mixed and sod grasses across the plains and lake basins, with poplars (*Populus* sp.) and willows (*Salix* sp.) along the river meadows (Esenov 1970).

### *Archaeological Background*

Eneolithic (Copper Age; second half of the 4<sup>th</sup> – first half of the 3<sup>rd</sup> mil. BC) and Early Bronze Age (second half of the 3<sup>rd</sup> – first quarter of the 2<sup>nd</sup> mil. BC) are generally the least understood periods in the history of Kazakhstan. Indeed, this was the time of transition to a producing economy and spread of metallurgy in the area. In the Early Bronze Age copper mining transformed into the use of artificial bronze alloys; ethno-cultural environment in the region changed dramatically.

The majority of studied sites of this period are located along the Ishim, Turgai and Irtysh Rivers. The Eneolithic sites of the middle Irtysh valley are represented by small poorly stratified settlements without a clear cultural attribution. As such, the discovery of the Borly 4 settlement is particularly important as this site has a potential of partly filling the existing archaeological “gap” in the region. The Early Bronze Age monuments in the region are represented by the sites of Yamnaya, Elunino, Afanasyevo, Chemurchek and other cultures. The two recently excavated sites of Shauke 1 and Shauke belong to the Elunino Culture. The pastoral European-type population of the Elunino Culture apparently was the first in the Ob-Irtysh interfluvium to practice bronze metallurgy (Grushin et al. 2009).

The economy of the populations is widely regarded as pastoral. Generally, the animal husbandry of Borly 4 is characteristic for the Eneolithic of the steppe zone of Kazakhstan (Gaiduchenko 2014). According to archaeozoological data, horse and cattle constituted more than 98% of the total meat component of the diet, with the rest (~1.7%) coming from wild game (including Przewalski-type horse, kulan, elk, wild boar and roe deer; Gaiduchenko and Merz 2012). In addition, birds and fish (small *Cyprinidae* specimens) were procured; the latter apparently being sourced from local reservoirs (excluding saline Lake Borly with brine shrimp *Artemia* the only fauna presented) (Uryvaev 1959) and possibly the Irtysh River. The pastoralism pattern of the Elunino Culture has been reconstructed as domestic-transhumant (Grushin 2013). The herd composition and the significance of particular species depended on environmental factors. For the steppe zone, horse was apparently the most significant source of meat, followed by cattle and then ovicaprids (despite the latter being the most numerous in

the herd); the herd composition was defined on the basis of the minimal number of individuals identified (Grushin 2013; Kiryushin 2002). Faunal remains also suggest hunting and fishing playing a supplementary role in the Elunino economy (ibid.).

To date, radiocarbon dates have only been obtained in the area for two Early Bronze Age sites of the Upper Irtysh River region (Kovalev 2009; Stöllner et al. 2013). As such, the Eneolithic and Early Bronze Age chronology of the region is vague and generally based on traditional archaeological dates. In this context, the use-time of particular monuments, as well as the internal periodization of different phases, are the main chronological issues of the area.

### *Palaeoeconomy Reconstructions in the Region using Stable Isotope Analysis*

Carbon ( $\delta^{13}\text{C}$ ) and nitrogen ( $\delta^{15}\text{N}$ ) stable isotope analysis has been used in palaeodietary and palaeoeconomy reconstructions for several decades (particularly, investigating the role of marine/freshwater component in the diet, as well as maize and millet consumption) and is currently one of the state-of-the-art techniques of modern bioarchaeology. Recently, research has expanded to understanding nuances of subsistence strategies and complex economic patterns of various prehistoric populations of the Eurasian Steppe (O'Connell et al. 2003; Privat et al. 2005; Shishlina et al. 2007, 2009; Svyatko et al. 2013; Murphy et al. 2013; Ventresca Miller et al. 2014). One of the topical and longstanding issues is the role of aquatic resources in their diet and economy, which is generally poorly understood, as very few associated archaeological remains have been recovered. Besides, the isotopic signatures of freshwater systems in Southern and Western Siberia and Kazakhstan are themselves not well-known. Traditional interpretations of the archaeological data for the prehistoric Eurasian Steppe populations have emphasized the pastoral element of the economy; however, recent bioarchaeological research suggests that fishing and gathering continued to be important. Through stable isotope studies, the importance of freshwater resources in the subsistence of the populations is being increasingly acknowledged, although the conclusions are complicated by the possibility of the aridity/salinity effect as one of the explanations of elevated  $\delta^{15}\text{N}$  values (e.g. Murphy et al. 2013; Ventresca Miller et al. 2014).

Methodological grounds of  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  stable isotope analysis are well described in present day literature. The analysis primarily concerns the protein part of the diet. Briefly, it is used to estimate the proportions of  $\text{C}_3$  versus  $\text{C}_4$  plants, proportion of marine and in some cases freshwater (e.g. Katzenberg and Weber 1999) components in the diet, and a trophic level of an individual.  $\text{C}_3$  plants (most plants) are well-adapted to temperate environments and are characterized by  $\delta^{13}\text{C}$  ca. = -26.5‰, while  $\text{C}_4$  plants (far less common, but including millet) are better adapted to higher aridity and temperature and demonstrate  $\delta^{13}\text{C}$  ca. = -12.5‰ (e.g. Pyankov et al. 2000; Wang et al. 2005; Makarewicz and Tuross 2006). The bone collagen from herbivores that subsist only on  $\text{C}_3$  grasses will give a  $\delta^{13}\text{C}$  value of ca. -21.5‰. If the diet were based only on  $\text{C}_4$  grasses then the value would be ca. -7.5‰. There is a small trophic level offset of 0.5-2‰ in  $\delta^{13}\text{C}$  for each step of a food chain (e.g. Bocherens and Drucker 2003; McCutchan et al. 2003). The  $\delta^{15}\text{N}$  values of most modern plants vary between 0 and 5‰, these increase on 3–6‰ with each step of a food chain (e.g. Hedges and Reynard 2007; O'Connell et al. 2012), and are most elevated when relying on aquatic resources. Non-dietary factors include the canopy effect (increase of leaf  $\delta^{13}\text{C}$  values from ground to canopy; van der Merwe and Medina 1991), climatic factors (higher plant  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  values and generally higher proportion of  $\text{C}_4$  grasses with increase in temperature and aridity; Ambrose 1991; van Klinken et al. 1994) and the manuring effect (increase of the  $\delta^{15}\text{N}$  ratios of manured soil and associated plants; Bogaard et al. 2013).

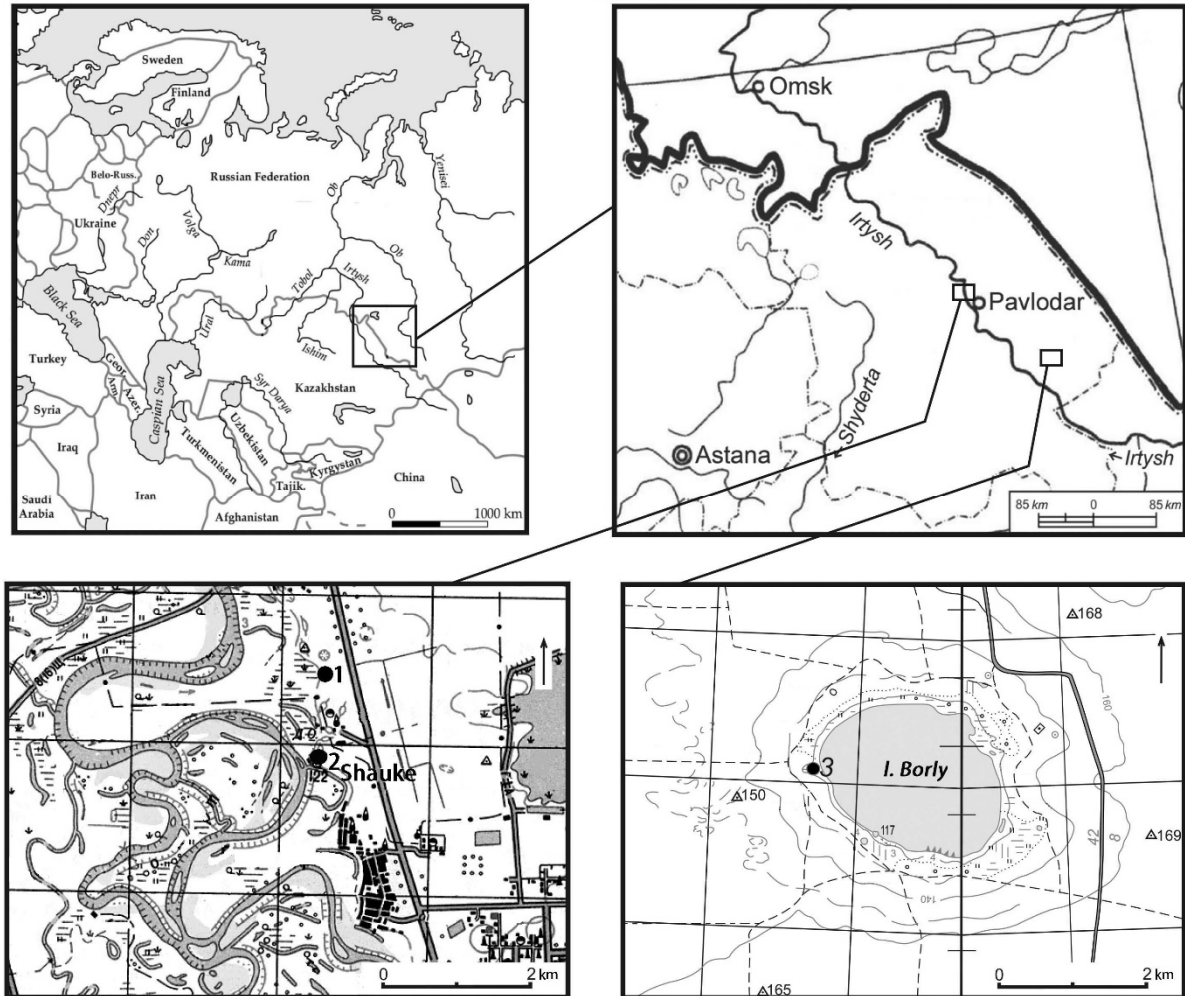
### *The Sites Analysed*

The present research involves isotopic and radiocarbon analysis of three sites – Borly 4, Shauke 1 and 8b, located in the middle course of the Irtysh River, on the territory of modern Pavlodar Oblast and excavated in 2010-2012 by V.K. Merz (Fig. 1). The cemetery of Shauke 1 (52°26'5.85"N 76°50'15.59"E) and the settlement of Shauke 8b (52°25'22.30"N 76°50'16.50"E) are situated near the modern village of Shauke on the sandy dunes of the fluvial terrace of the Irtysh River right shore 10 km north from the city of Pavlodar. The settlement of Borly 4 (51°49'28.9"N 77°56'42.4"E) is located 35 km east from the river, on the western terrace above the Lake Borly flood-plain.

The multi-layer settlement of Borly 4 has a total area of 175 m<sup>2</sup>. The site is well stratified and contains archaeological materials from Middle Neolithic to Early Iron Age, however, the main cultural layer belongs to the Eneolithic period. The latter includes the remains of dwellings and household outbuildings containing a large number of stone, ceramic and copper objects, together with horse and cattle bones. The obtained materials indicate a well-developed producing economy (Gaiduchenko and Merz 2012; Gaiduchenko 2013), which, coupled with a distinctive material culture, suggest the existence of a particular Borly Culture.

The cemetery of Shauke 1 is located in a sand quarry and consists of 5 graves spread in the area of 2100 m<sup>2</sup>. Four graves contained disarticulated human and animal bones, pottery, and bronze and stone objects. The fifth tomb apparently represented a cenotaph as no human remains were found; cremated animal bones and charcoal were recovered from the filling of the grave. The site is now completely destroyed. The artefacts from the site have similarities to those from the Elunino sites of Steppe Altai and North-East of Kazakh Uplands (Merz 2008; Grushin 2013), however specific features of Shauke 1 (unusual artefacts such as a stone axe and foundry ladle, the presence of arsenic in bronze alloys, specifics of pottery and its similarity with that of Western Catacomb cultures) suggest its earlier chronological position.

The settlement of Shauke 8b is a single layer site of the Elunino Culture of a total area of 54 m<sup>2</sup>. The average thickness of the cultural layer is 10-15 cm; the southern edge of the site was destroyed by a sand quarry. The cultural layer was overlaid by a two-meter dune containing a 20 cm thick palaeosol. The northern part of the cultural layer was destroyed by wind erosion. The settlements yielded remains of fireplaces, ash pits, faunal (including fish) bones, pottery, rare stone and metal artefacts, and copper ore. A particular feature of the site is the presence of large quantities of fish bones. No dwelling structures were found – apparently, the settlement represented a seasonal camp. The artefacts found have similarities with those of the Elunino Culture. Single layer sites as Shauke 8b are very rare in the region and can be used to refine the internal chronology of archaeological cultures.



**Fig. 1. Provenance of test specimens: 1. Shauke 1; 2. Shauke 8b; 3. Borly 4.**

## Materials and Methods

For correcting the  $^{14}\text{C}$  dates of human bones, the FRE can be calculated for the region by measuring contemporaneous terrestrial and freshwater samples and the proportion of aquatic diet in the humans is assessed by stable isotope measurements. As such, this study involves two major techniques:

a) AMS  $^{14}\text{C}$  dating of synchronous archaeological samples (e.g. freshwater fish and/or human versus terrestrial herbivores or associated plant/charcoal samples) from strictly the same archaeological context. The difference in  $^{14}\text{C}$  ages within these pairs/groups will indicate the extent of the freshwater reservoir offset (FRO) in the area and help to quantify the uncertainty in radiocarbon ages of human samples where  $\delta^{15}\text{N}$  values indicate freshwater dietary input.

b) carbon and nitrogen ( $\delta^{13}\text{C}$ ,  $\delta^{15}\text{N}$ ) stable isotope analysis of bone collagen on archaeological samples for dietary and isotopic background assessment, in particular – for estimation of the proportion of non-terrestrial material in the diet of the analysed individuals.

## Materials

In total, eleven groups of associated samples (n=32) from three sites have been analysed, including four human, 26 animal (five of them are calcined; seven of them are fish), and two charcoal samples (Table 1). One group from Shauke 8b did not include associated aquatic and human samples, however it was incorporated in the study for consistency of the archaeological chronological and isotopic palaeoenvironmental record.

### *Pretreatment of samples*

Elemental Analysis - Isotope Ratio Mass Spectrometry (EA-IRMS)  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  isotope measurements and AMS  $^{14}\text{C}$  dating of samples were performed in the  $^{14}\text{C}$ CHRONO Centre for Climate, the Environment and Chronology (Queen's University Belfast).

For bone samples, the surfaces were cleaned using a brush prior to collagen extraction. Extraction of collagen from archaeological samples was based on the ultrafiltration method (Brown et al. 1988; Bronk Ramsey et al. 2004) with an additional sodium hydroxide (NaOH) wash step (Brock et al. 2010). Briefly, these included:

- a) bone demineralization in 2% HCl, followed by MilliQ® ultrapure water wash;
- b) 0.1M NaOH treatment for 15 min, followed by MilliQ® wash;
- c) 2% HCl wash for 15 min, followed by MilliQ® wash;
- d) gelatinization in weak HCl at 58°C for 16 hours;
- e) filtration, using ceramic filter holders, glass filter flasks and 1.2  $\mu\text{m}$  glass microfiber filters;
- f) ultrafiltration using Vivaspin® 15S ultrafilters with MWCO 30 kDa; 3000-3500 rev/min for 30 minutes, and
- g) freeze-drying; the dried collagen was stored in a desiccator.

Being extremely fragile and in most cases in very small samples, archaeological fish bones were not subjected to NaOH treatment to avoid further collagen loss, as NaOH treatment has shown to decrease collagen yields of bone samples (Liden et al. 1995).

To explore the possible effect of the NaOH treatment on stable carbon and nitrogen isotope ratios of bone collagen, duplicates were prepared for 13 bone samples following the ultrafiltration method (Brown et al. 1988; Bronk Ramsey et al. 2004), and omitting the NaOH wash step.

Pretreatment of charcoal was performed following the standard AAA procedure (Mook and Waterbolk 1985) and included, 2% NaOH wash for 2 hrs followed by another 4% HCl wash on an 80°C hotplate for 2-3 hrs.

Cremated bone pre-treatment procedure followed Lanting et al. (2001) and included bone crushing, 1.5% sodium hypochlorite ( $\text{NaClO}$ ) wash for 48 hrs, 1M acetic acid ( $\text{C}_2\text{H}_4\text{O}_2$ ) wash for 24 hrs, vacuum filtration and drying overnight, followed by combustion with silver.

### *$\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ stable isotope analysis and AMS radiocarbon dating*

$\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  of bone gelatin were measured on a Thermo Delta V Isotope Ratio Mass Spectrometer (IRMS) with Thermo Flash 1112 Elemental Analyser (EA) peripheral. The measurements were made in duplicate. Results were reported using the delta convention relative to international standards – VPDB for C and AIR for N (Hoefs 2009). The measurement uncertainty (1sd) of  $\delta^{13}\text{C}$ ,  $\delta^{15}\text{N}$  and  $\text{C:N}_{\text{atomic}}$  based on 6-10 replicates of 7 bone collagen samples is 0.22‰, 0.15‰ and 0.2, respectively.



With the exception of fish bone samples, only bone samples subjected to NaOH wash were measured for AMS  $^{14}\text{C}$ . Prepared bone collagen and plant samples were sealed under vacuum in quartz tubes with an excess of CuO and combusted at 850°C. The CO<sub>2</sub> was converted to graphite on an iron catalyst using a zinc reduction method (Slota et al. 1987). The graphite was then pressed to produce a “target” and this was measured by 0.5 MV National Electrostatics Compact AMS. The  $^{14}\text{C}$  age and one standard deviation were calculated using the Libby half-life (5568 years) following the conventions of Stuiver and Polach (1977). The radiocarbon ages were then corrected for isotopic fractionation using the AMS-measured  $\delta^{13}\text{C}$ . The radiocarbon ages were calibrated using Calib 7.0 programme (Stuiver et al. 2013) and IntCal13 calibration curve (Reimer et al. 2013).

Statistical analysis (linear correlations, two-tailed paired t-tests, significance level of 0.05) was performed using Microsoft Office Excel 2013.

### *Calculating the freshwater reservoir offset (FRO)*

Two ways of calculating FRO for the cases where more than two associated samples are compared (represented here by four groups of samples from Shauke 8b) have been used. The first approach is to combine the dates for the terrestrial samples using uncertainty-weighted means and then use this pooled  $^{14}\text{C}$  date for a comparison with the aquatic sample (fish). Prior to combining the radiocarbon dates, their statistical proximity was assessed using the Ward and Wilson (1978) chi-squared test in Calib 7.0. The FRO uncertainty is the sum of the squares of the uncertainty in the terrestrial and aquatic  $^{14}\text{C}$  values. An alternative, second, approach for calculating the FRO is to calculate a separate offset for each terrestrial sample vs. fish within the groups, and then combine these individual offsets using uncertainty-weighted means. In this case, the uncertainty is taken as the square root of the variance of the offsets. Table 1 presents the FRO values calculated using both ways.

## **Results and Discussion**

### *Preservation of bone samples*

The majority of bone samples analyzed demonstrated very good collagen preservation with its content varying within 4.3-16.4% (van Klinken 1999; Table 1). Three samples from Shauke 8b and Borly 4 yielded no collagen, and one fish sample from Borly 4 (UBA-27721) yielded very low collagen content – only 0.7%. The collagen content of this latter sample (UBA-27721) is rather marginal and, as suggested by van Klinken 1999, “might or might not be suitable for analysis”. We include this sample into the discussion of the results, however we do admit that the interpretation for this sample is rather ambiguous (see below). The C:N<sub>atomic</sub> ratio of the samples varied between 3.1-3.4, which is also within the accepted range characterizing a well-preserved collagen (DeNiro 1985).

**Table 1. Results of AMS <sup>14</sup>C dating, stable C and N isotope analysis of the samples and calculated freshwater reservoir offsets (FRO)**

Radiocarbon calibration is discussed in the text

\*Calibrated <sup>14</sup>C dates from aquatic and human samples need to be interpreted with caution as might require a FRO correction

Lab ID	Sample	Provenance	<sup>14</sup> C BP	cal BC (2 σ)	Combined terrestrial <sup>14</sup> C age BP	FRO with combined terrestrial <sup>14</sup> C BP	FRO	Combined FRO	NaOH treatment applied				No NaOH treatment			
									% coll.	δ <sup>13</sup> C	δ <sup>15</sup> N	C:N <sub>at</sub>	% coll.	δ <sup>13</sup> C	δ <sup>15</sup> N	C:N <sub>at</sub>
Shauke 8b (52°25'22.30"N 76°50'16.50"E)																
UBA-26189	fish	sq. A1, hearth 2	3802±37	2435-2063 BC*	-	269±69	-	260±61	-	-	-	-	3.3	-24.3	8.7	3.4
UBA-26190	herbivore		3624±48	2136-1884 BC	3533±59		178±61		n/a	-18.6	6.9	3.2	16.2	-19.2	7.9	3.2
UBA-26191	charcoal		3532±34	1948-1754 BC			270±50		-	-	-	-	-	-	-	-
UBA-26192	animal, calcined		3501±29	1905-1744 BC			301±47		-	-	-	-	-	-	-	-
UBA-26197	fish	sq. A3	3771±39	2333-2038 BC*	-		-		-	-	-	-	1.7	-22.5	9.7	3.2
UBA-26198	ovicaprid		3603±42	2130-1784 BC	3614±16	157±42	168±57	157±16	9.7	-18.4	7.2	3.2	5.7	-18.5	7.2	3.2
UBA-26199	animal, calcined		3626±43	2134-1888 BC			145±58		-	-	-	-	-	-	-	-
UBA-26200	fish	sq. a I	3775±42	2339-2038 BC*	-		-		-	-	-	-	3.6	-24.2	8.8	3.3
UBA-26201	herbivore		3678±41	2196-1945 BC	3614±74	161±85	97±59	154±74	12.5	-18.7	8.4	3.2	10.5	-18.9	6.4	3.2
UBA-26202	animal, calcined		3573±33	2025-1780 BC			202±53		-	-	-	-	-	-	-	-
UBA-26193	fish	sq. A 7, ash pit	-	-	-	-	-	-	0	-	-	-	-	-	-	-
UBA-26194	herbivore		-	-	-	-	-	-	0	-	-	-	-	-	-	-
UBA-26203	fish	sq. A II, pit	3810±43	2456-2137 BC*	-		-		-	-	-	-	1.8	-22.9	9.4	3.2
UBA-26204	ovicaprid		3571±36	2027-1777 BC	3586±19	224±47	239±56	225±19	5.7	-18.6	7.1	3.2	4.8	-18.1	7.1	3.2
UBA-26205	animal, calcined		3598±32	2033-1884 BC			212±54		-	-	-	-	-	-	-	-
Shauke 1 (52°26'5.85"N 76°50'15.59"E)																
UBA-26890	human	grave 1	3763±34	2289-2042 BC*	-	53±53	-	-	4.3	-19.2	13.3	3.2	7.4	-19.2	13.4	3.2
UBA-26891	ovicaprid/deer		3710±41	2271-1976 BC	-		-	-	12.2	-20.2	9.8	3.2	10.4	-20.1	9.9	3.2
UBA-26892	human	grave 2	3772±33	2293-2047 BC*	-	66±49	-	-	9.6	-19.2	13.5	3.2	2.0	-19.5	13.4	3.2
UBA-26893	sheep		3706±36	2202-1980 BC	-		-	-	9.7	-19.3	6.8	3.2	13.0	-19.3	6.9	3.1
UBA-26894	human	grave 3	3782±33	2334-2050 BC*	-	21±52	-	-	3.7	-18.4	14.1	3.2	8.8	-18.5	14.1	3.2
UBA-26895	sheep		3761±40	2292-2036 BC	-		-	-	4.3	-18.6	8.0	3.2	2.3	-18.8	8.0	3.2
UBA-26896	human	grave 4	3883±37	2470-2212 BC*	-	92±55	-	-	6.7	-19.3	13.8	3.2	9.9	-19.4	13.5	3.1
UBA-26897	sheep, juvenile		3791±40	2427-2046 BC	-		-	-	6.5	-19.4	7.8	3.2	14.9	-19.8	7.1	3.2
UBA-26898	charcoal	grave 5	3863±35	2463-2209 BC	-	n/a	-	-	-	-	-	-	-	-	-	-
UBA-26899	deer/ovicaprid, calcined		3883±32	2468-2234 BC	-		-	-	-	-	-	-	-	-	-	-
UBA-26900	horse/cattle		104±51	1675-1941 AD	-		-	-	16.4	-20.5	5.1	3.2	10.1	-20.5	4.9	3.1
Borly 4, house 3 (51°49'28.9"N 77°56'42.4"E)																
UBA-27719	horse	main bone-bearing horizon in the ash layer	4387±34	3095-2912 BC	-	-	-	-	11.2	-19.6	6.2	3.3	-	-	-	-
UBA-27720	cattle		4507±41	3360-3037 BC	-	-	-	-	4.9	-18.8	8.0	3.2	-	-	-	-
UBA-27721	fish		4241±46	2925-2666 BC*	-	-	-	-	0.7	-	-	-	-	-	-	-
UBA-27722	fish		-	-	-	-	-	-	0	-	-	-	-	-	-	-
UBA-27723	dog	sq. B-I, layer 3a	4607±39	3517-3125 BC*	-	-	-	-	12.6	-21.2	11.4	3.2	-	-	-	-

UBA-27724	fox	sq. B-I, 53-56 cm	4346±34	3083-2896 BC	-	-	-	-	12.1	-18.6	8.7	3.3	-	-	-	-
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### *The effect of NaOH treatment on bone collagen $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$*

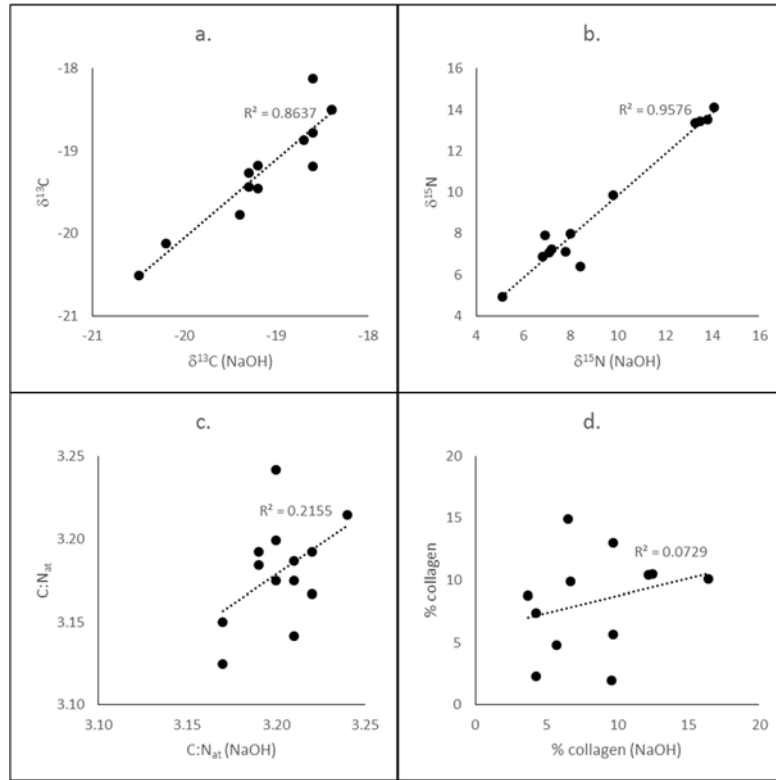
NaOH wash is often used in radiocarbon sample preparation to remove humic acids as one of the main contaminants of the bone collagen (Berglund et al. 1976; DeNiro and Epstein 1981; Brock et al. 2010), however, there have been many concerns regarding the possibility of preferential affecting specific amino acids and, as a result, alteration of overall  $\delta^{13}\text{C}$  signal of bone collagen (see discussion in Liden et al. 1995). Recent data demonstrated minor but systematic alteration of  $\delta^{13}\text{C}$  values of the samples as a result of NaOH wash (Jorkov et al. 2007), however, in this case, lower  $\delta^{13}\text{C}$  of bone collagen samples which have not been subjected to NaOH wash were explained as possibly affected by preservation of some lipids, known to have more negative  $\delta^{13}\text{C}$  values compared to those of protein. This study also demonstrated no systematic effect of NaOH wash on  $\delta^{15}\text{N}$  values of the samples, slightly lower C:N atomic ratios compared to the samples treated with ultra-filtration method (Brown et al. 1988 modified in Richards and Hedges 1999; includes both ultra-filtration and filtration with Eze® filter separators), and fairly consistent %C and %N values of 41.1-44.7% and 14-16% respectively, suggesting that the use of NaOH removes the non-protein contaminants. The data on the effect of NaOH treatment on collagen yields of bone samples generally demonstrates a decrease in collagen yields (Liden et al. 1995) although not as remarkable as the effect of ultrafiltration (Jorkov et al. 2007).

In our study, 13 samples have been prepared in duplicate to further explore the possible effect of NaOH treatment at the concentrations used for the  $^{14}\text{C}$  pretreatment on stable  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  isotope ratios, C:N<sub>atomic</sub> and collagen yields (for % collagen comparison, only twelve pairs were available). Our t-test results at a significance level of 0.05 indicate a minor but significant effect of NaOH only for C:N<sub>atomic</sub> ratios of the samples ( $p=0.014$ ; Table 2) – samples treated with NaOH appear on average 0.02 higher than non-treated.

**Table 2. Comparative analysis of the differences in  $\delta^{13}\text{C}$ ,  $\delta^{15}\text{N}$ , C:N<sub>atomic</sub> and collagen yields between the samples pretreated with and without NaOH wash step**

Testing	N of observations	Mean±1sd (NaOH-treated)	Mean±1sd	<i>t</i>	<i>p</i>
$\delta^{13}\text{C}$ , ‰	13	-19.1±0.6	-19.2±0.6	1.429	0.179
$\delta^{15}\text{N}$ , ‰	13	9.4±3.0	9.2±3.1	0.823	0.427
C:N <sub>atomic</sub>	13	3.20±0.0	3.18±0.0	2.880	<b>0.014</b>
% collagen	12	8.4±3.8	8.3±3.8	0.090	0.930

Despite the presence of several outliers, generally  $\delta^{13}\text{C}$  or  $\delta^{15}\text{N}$  isotope ratios demonstrate significant linear correlation between the values of samples pretreated in different ways ( $R^2=0.86$  for  $\delta^{13}\text{C}$ ;  $R^2=0.96$  for  $\delta^{15}\text{N}$ ; Fig. 2). No significant correlation has been observed for the C:N<sub>atomic</sub> levels of the samples ( $R^2=0.22$ ), however, in both cases the values for the prepared gelatin range between 3.12 and 3.24 which indicates a very high degree of consistency. Collagen yields varied between 3.7% and 16.4% for samples subjected to NaOH wash, and between 2.0% and 14.9% for those not subjected to NaOH treatment. No significant correlation has been observed in this case ( $R^2=0.07$ ).



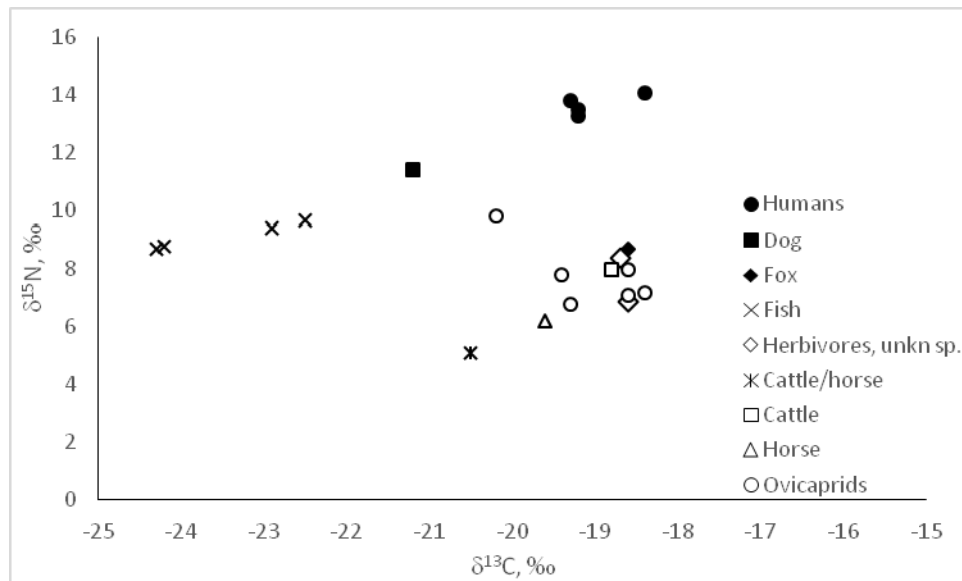
**Fig. 2. Plot showing the correlation between  $\delta^{13}\text{C}$  (a) and  $\delta^{15}\text{N}$  (b) isotope ratios, C:N<sub>atomic</sub> levels (c) and collagen yields (d) of samples subjected to NaOH treatment and their NaOH wash-free duplicates**

Further in the text, the discussion only concerns bone samples pre-treated using NaOH (with the exception of fish samples).

#### *$\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ stable isotope analysis – palaeodietary implications*

The study represents the first attempt to assess the human subsistence patterns for the north-eastern Kazakhstan prehistoric populations. Unfortunately precise species determinations were not available for a number of faunal samples, which complicates some aspects of interpretation of the results. Given a limited number of samples, and general geographical and environmental similarity of the sites analysed, the samples were plotted and discussed together.

In total one dog, one fox, four fish, eleven domestic herbivore, and four human samples have been analysed for stable isotope ratios. Overall, the results do not suggest major variations within these groups and indicate the C<sub>3</sub>-based foodchain with no evidence of C<sub>4</sub> plant consumption (such as millet; Fig. 3).



**Fig. 3. Bone collagen  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  values for human and faunal samples included in the study (n = 21)**

The mean  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  values for freshwater fish (n=4) from the settlement of Shauke 8b are  $-23.5 \pm 0.9\text{‰}$  and  $9.2 \pm 0.5\text{‰}$  respectively. The low carbon isotopic values are typical for the inland freshwater reservoirs characterized by primarily  $\text{C}_3$  foodchains and depleted  $\delta^{13}\text{C}$  in the local fauna (unlike e.g. Lake Baikal ecology, Katzenberg and Weber 1999).

There are two outliers among the analysed herbivores (n=11). The horse/cattle sample from grave 5 of Shauke 1 (UBA-26900) clearly demonstrated the lowest  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  values among other herbivores analysed ( $-20.5\text{‰}$  and  $5.1\text{‰}$  resp.). As this sample is dated to 1675-1941 cal AD, there is a possibility that it has been affected by modern changes in the atmospheric reservoir due to fossil fuel burning ("Suess effect") although the  $\delta^{13}\text{C}$  depletion from pre-industrial value of  $-6.5\text{‰}$  until AD 1940 has only been  $0.3 - 0.4\text{‰}$  (Francey et al. 1999). We could also suggest that, belonging to a different period and culture, that animal was subjected to a different pasture strategy (e.g. in a more forested environment). Another clear outlier is an ovicaprid from grave 1 of Shauke 1 (UBA-26891) which has the highest  $\delta^{15}\text{N}$  values among herbivores analysed ( $9.8\text{‰}$ ) and second lowest  $\delta^{13}\text{C}$  values ( $-20.2\text{‰}$ ). At the moment it is not clear what could cause such unusual isotopic values. One of the suggestions would be the use of manured fodder (pasture) by this animal, as this could significantly increase bone collagen  $\delta^{15}\text{N}$  ratios of the analysed individual. However, this hypothesis needs to be tested against a larger number of herbivorous samples from the same culture. With the exclusion of the two outlying individuals, the analysed herbivores (n=9) have mean  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  values of  $-18.9 \pm 0.4\text{‰}$  and  $7.4 \pm 0.7\text{‰}$  respectively.

The isotopic values of a single fox analysed ( $\delta^{13}\text{C} = -18.6\text{‰}$ ;  $\delta^{15}\text{N} = 8.7\text{‰}$ ) are only  $0.3\text{‰}$  higher in  $\delta^{13}\text{C}$  and  $1.3\text{‰}$  higher in  $\delta^{15}\text{N}$  than the associated mean values for herbivores. This difference is smaller than commonly observed trophic level offset of  $0.5\text{‰}$ - $2\text{‰}$  for  $\delta^{13}\text{C}$  and  $3\text{‰}$ - $6\text{‰}$  for  $\delta^{15}\text{N}$  (see Introduction), however, at the moment no local wild herbivorous species (such as steppe rodents, reptiles or birds which would constitute the diet of a steppe fox) have been analysed and as such we cannot make conclusions on the isotopic signature of this animal's diet.

All human samples analysed (n=4) come from the cemetery of Shauke 1. The mean  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  values for the humans are  $-19.0 \pm 0.4\text{‰}$  and  $13.7 \pm 0.4\text{‰}$  respectively, which indicates

a diet based predominately on C<sub>3</sub> foodchains and animal protein. Isotopically, humans cluster together quite tightly which indicates the absence of major variation in their diet. Human mean  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  ratios are respectively 0.1‰ and 6.3‰ higher than those of herbivores analysed. The human-herbivore nitrogen offset is slightly larger than the commonly observed trophic level increase of 3–6‰ and suggests that herbivores may not represent the only source of dietary protein for humans, however their diet may have also included fish which is at a slightly higher trophic level than terrestrial herbivores analysed due to the multiple steps in an aquatic foodweb. Similar conclusion could be drawn regards the diet of a single dog analysed – its isotopic signature ( $\delta^{13}\text{C} = -21.2\text{‰}$ ;  $\delta^{15}\text{N} = 11.4\text{‰}$ ) suggests the contribution of both domestic herbivores and fish into the diet. However, the amount of fish in the dog's diet was possibly slightly larger compared to human diet as canine  $\delta^{13}\text{C}$  are closer to that of fish.

Recently, there has been a number of reports suggesting, that, despite the traditional perception of steppe communities as pastoralists with similar herd-based economies, apparently, depending on local climate, topography and ecology, there was variation in subsistence strategies between the groups, with some of them relying on fishing and/or cultivation (e.g. Frachetti et al. 2010; Murphy et al. 2013). Archaeologically, fishing is regarded as playing only a minor role in the economy of the Early Bronze Age populations in the region, as very few associated artefacts have been recovered. However, the discovery of a large quantity of fish remains in the settlement of Shauke 8b, as well as current results of stable isotope analysis on humans from Shauke 1, suggest the opposite – fishing was an accounted economical feature for this population. Undoubtedly, more samples, in particular human, need to be analysed to draw more definite conclusions on the extent of fishing for various prehistoric communities of the area.

The majority of the samples analysed for stable isotopes during the current study were fauna, which contributes to the reconstruction of the isotopic baseline for the region. Generally, the results indicate primarily C<sub>3</sub>-based ecology (both terrestrial and aquatic) of the Pavlodar area, and also suggest the absence of millet cultivation in the area in the Middle Bronze Age (the latter conclusion is however based on extremely small number of human samples analysed). Our data generally correspond with results for a number of Middle to Late Bronze Age sites in Northern (Ventresca Miller et al. 2014) and Central Kazakhstan (Lightfoot et al. 2014), and Baraba forest-steppe of south-western Siberia (Privat et al. 2005) which isotopically document the appearance of millet consumption in the Central Kazakhstan only in the Late Bronze Age (Lightfoot et al. 2014).

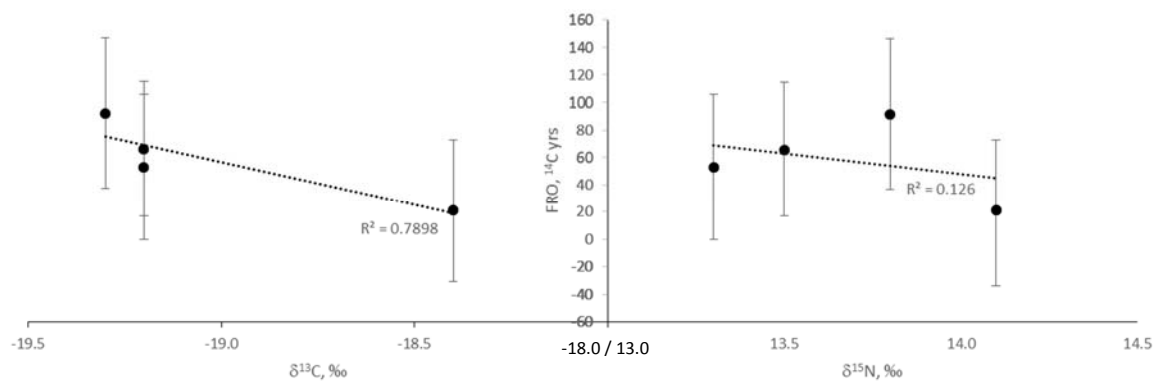
#### *AMS radiocarbon dating results – FRO*

Ten associated human - terrestrial herbivore or fish - terrestrial plant/herbivore groups from all three sites have been AMS radiocarbon dated to assess the freshwater reservoir offset (FRO) at the Pavlodar area. Unfortunately, precise fish species determinations were not available. The group from house 3 of Borly 4 settlement demonstrated quite variable  $^{14}\text{C}$  age ranges of samples, which apparently represent several phases of use of the building (see discussion in the next section) and, as such, they apparently cannot be strictly associated with each other as suggested before the analysis, and used for the estimation of the FRO.

For other groups, FRO was calculated using the two methods described above. For the first method, in all cases apart from samples UBA-26201 and UBA-26202 (which might possibly be explained by the small number of terrestrial samples analyzed) the combined dates from terrestrial samples were statistically the same at 95% level (using the Ward and Wilson (1978) chi-squared test in Calib 7.0). In all cases, the resulting final FROs for the four groups

calculated using the two methods show very little difference. In general, for Shauke 8B FROs between fish and individual terrestrial samples range from  $97 \pm 59$  to  $301 \pm 47$   $^{14}\text{C}$  yrs; the overall mean FRO (calculated by combining all individual offsets of terrestrial samples versus fish using uncertainty-weighted means) is  $210 \pm 65$   $^{14}\text{C}$  yrs.

Human - terrestrial herbivore pairs from Shauke 1 show less variation in FRO, which is not unexpected as the diet of human individuals would only partly be based on aquatic resources. As suggested above, the diet of the human individuals possibly included proportions of fish. The FRO values within the pairs vary from  $21 \pm 52$  to  $92 \pm 55$   $^{14}\text{C}$  yrs; the general mean FRO for Shauke 1 (calculated by combining all individual human - terrestrial herbivore offsets using uncertainty-weighted means) is  $57 \pm 29$   $^{14}\text{C}$  yrs. The results for the human samples suggest negative correlation between FROs and  $\delta^{13}\text{C}$  values ( $R^2=0.790$ ;  $n=4$ ; Fig. 4), which is to be expected with more depleted (negative)  $\delta^{13}\text{C}$  of fish yielding higher FRO values. This correlation is the strongest evidence for freshwater resources in the diet for these individuals. In comparison, no correlation was observed between human FRO and  $\delta^{15}\text{N}$  values, which is possibly not surprising given only 1.8‰ difference between mean  $\delta^{15}\text{N}$  values of fish and herbivores (unlike 4.4‰ difference in  $\delta^{13}\text{C}$ ). Furthermore, the human with the highest  $\delta^{15}\text{N}$  value (14.1‰) also has the lowest offset value with the associated herbivore sample ( $21 \pm 52$   $^{14}\text{C}$  yrs).



**Fig. 4. Linear regression plot of human  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  values and human-faunal  $^{14}\text{C}$  offsets (means and sd)**

The results obtained represent the first data for the freshwater offset in Kazakhstan, as such; no comparative material is available for the area at the moment. The geographically closest research, undertaken on Lake Baikal region, has the highest offset of  $\sim 700$   $^{14}\text{C}$  yrs for the endemic seals in the lake (Nomokonova et al. 2013), while in humans the highest detected offset equals 622  $^{14}\text{C}$  yrs (Schulting et al. 2014). Such a large value for the freshwater reservoir offset in the area is related to the nature of the lake itself, being the largest, deepest, and oldest freshwater lake in the world. However, there is still an ongoing discussion on the particular factors affecting the extent of the offset (ibid.). More distantly, on North Caucasus site of Klin-Yar, the offset values between humans and historically dated artefacts have been found highly variable, ranging between 0 to  $\sim 350$   $^{14}\text{C}$  yrs (Higham et al. 2010). Even more distantly, at the Dnieper Rapids area, the FRO values for human-faunal/fish paired samples vary between 125-750  $^{14}\text{C}$  yrs (Lillie et al. 2009). Among these three studies, only for the Lake Baikal region was it possible to calculate the correction to account for the FRO in humans as  $77 \pm 10$   $^{14}\text{C}$  yrs for each per mil increase in  $\delta^{15}\text{N}$  (Schulting et al. 2014).



The FRO values observed in our study (maximum fish-herbivore offset of  $301 \pm 47$   $^{14}\text{C}$  yrs) generally correspond with the data above, although the highest values are much lower than in Baikal and Dnieper Rapids regions which is apparently due to a smaller proportion of dissolved old carbon in the water.

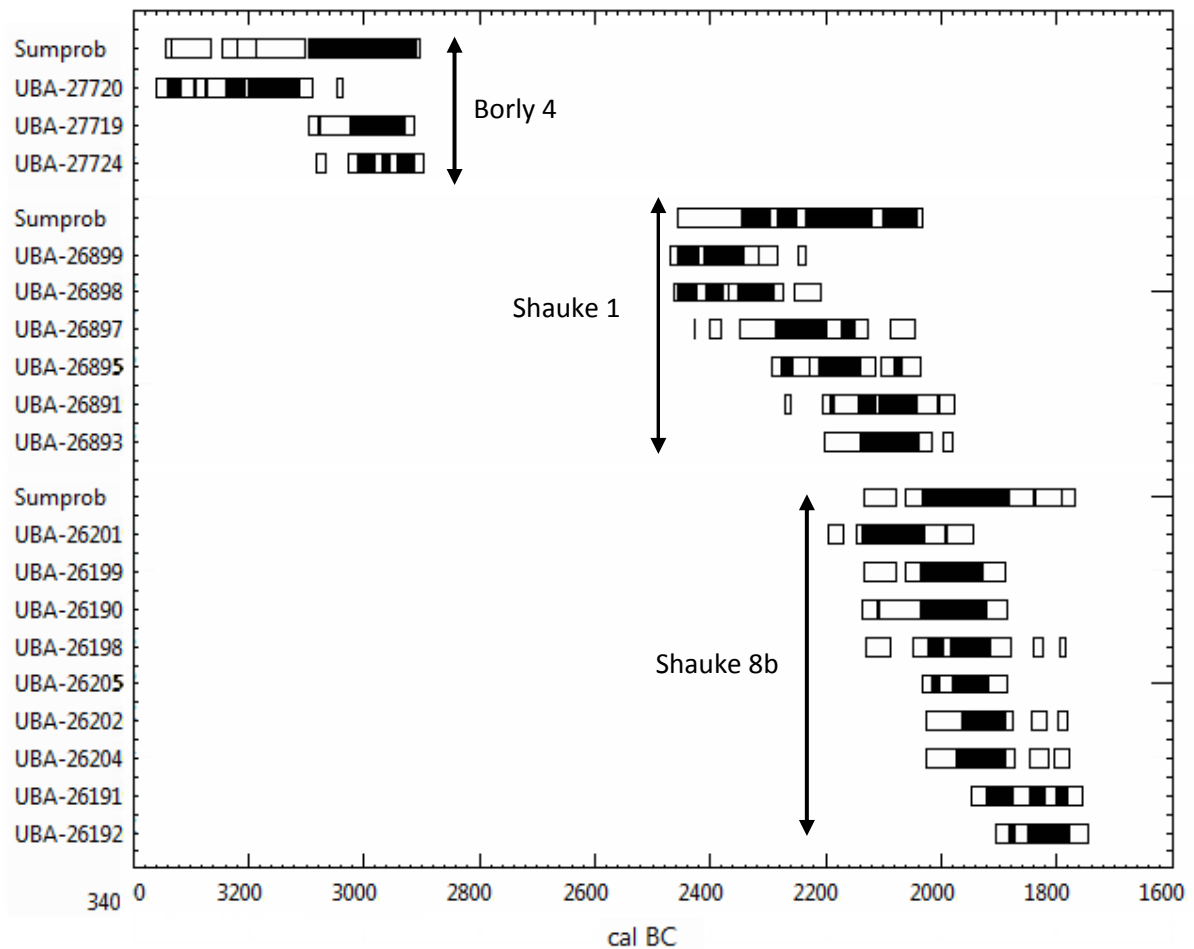
More human samples need to be tested for  $^{14}\text{C}$  and stable isotope values to assess the FRO correction for the area.

#### *AMS radiocarbon dating results – archaeological implications*

In total, 28 dates have been obtained for the three sites analysed. The horse sample from Shauke 1, dated to a modern period (UBA-26900; 1680-1940 cal AD), is obviously intrusive in the burial and therefore is not discussed further in the text.

The group from Borly 4 settlement demonstrated quite variable  $^{14}\text{C}$  age ranges of samples, with cattle and dog samples being the oldest (3360-3040 cal BC and 3520-3130 cal BC respectively) and the fish sample being the youngest (2930-2670 cal BC). Being an “open” complex (unlike “closed” complexes such as graves), the assemblage of house 3 apparently represents several phases of use— as a living space (a house) and, apparently some time later, as a midden (the midden was likely filled during a very short period of time as no signs of weathering have been observed on bones). At the moment we can suggest that the older date of the dog sample is likely to be affected by the FRO rather than actually belonging to an earlier period of use of the building, as the bone was stratigraphically located above the cattle sample in the midden. However, the cattle sample bone analysed was recovered from the very bottom of the midden and therefore there is a chance that it could have belonged to the earlier phase of the site functioning. The interpretation of the  $^{14}\text{C}$  date of the fish sample is rather complicated. The sample yielded very low % collagen (0.7%), which might be an indication of collagen degradation. It is also possible that contaminating humic acids were not removed since no NaOH step was used. However, the date can also be interpreted as associated with the later Early Bronze Age phase of the site – the existence of multiple burrows suggests that that bone could have been relocated by animals. Furthermore, we cannot deny the possibility of FRO affecting the date of fish sample (i.e. the actual date of this sample being even younger). There is no direct evidence for the latter suggestion, however, there is a chance that the fish might have been taken from the Irtysh River and then might be bearing an associated offset.

In general, dates obtained from fish, dog and human bone samples are not included in the discussion below as they are likely to be affected by the FRO (see section above). The remaining 18 dates cluster well together within the sites (Fig. 5). The  $2\sigma$  summed probability age ranges are 3340-2900 cal BC for Borly 4, 2470-2230 cal BC for Shauke 1, and 2130-1770 cal BC for Shauke 8b.



**Fig. 5. Calibrated age ranges of the samples analysed (dates obtained from fish, dog and human bone samples are not included as likely to be affected by the FRO)**

The obtained AMS dates are the first for the Eneolithic of the Middle and Upper Irtysh River region and correspond with the archaeological chronology for the sites. The new  $^{14}\text{C}$  dates of the Borly 4 settlement suggest that the site is generally synchronous to the Eneolithic sites of South-Western Siberia and Kazakhstan, including Novoiyinka 3 and 6 in the North Kulunda River area (38<sup>th</sup>-25<sup>th</sup> c. BC), sites of the Ust-Narym-Shiderty type in the North-East Kazakh Uplands (second half of the 4<sup>th</sup> – first half of the 3<sup>rd</sup> mil. BC; Merz 2008; Gaiduchenko and Kiryushin 2013), Tersek Culture settlements of Kozhai 1 and Kumkeshu 1 in Turgai region (41<sup>st</sup>-25<sup>th</sup> c. BC; Kaliyeva and Logvin 1997), Botai Culture settlements of Botai and Krasny Yar in the forest-steppe Ishim River region (38<sup>th</sup>-32<sup>nd</sup> c. BC; Levine and Kislenko 2002), as well as to the Afanasyevo Culture sites of the Altai Mountains and Yenisei River (38<sup>th</sup>-25<sup>th</sup> c. BC), and Yamnaya Culture sites of the Volga-Ural region (39<sup>th</sup>-27<sup>th</sup> c. BC; Polyakov 2010; Morgunova 2014). Archaeologically, the above sites reveal similar economical features characteristic to early pastoral societies (Gaiduchenko 2013; Gaiduchenko and Kiryushin 2013). At this stage, it can be argued that the sites of the Botai and Tersek Cultures are earlier than Borly 4 and possibly Novoiyinka 3 and 6, however, we can suggest that, as a result of further research, the chronological dates of the Borly 4 settlement and generally the Eneolithic period of the region will be expanded, possibly towards earlier period.

The Early Bronze Age period of the Middle Irtysh River region is mainly represented by the Elunino Culture which is radiocarbon dated to the 25<sup>th</sup>-18<sup>th</sup> c. BC (Grushin 2013). Previously, the earliest Elunino sites have only been found north-east of Sary-Arka (Kazakh Uplands, to the west from Irtysh River) and radiocarbon dated to 25<sup>th</sup>-22<sup>nd</sup> c. BC (*ibid.*). The dates obtained from the Shauke 1 cemetery suggest that the site was used for extended period of time. The two earlier graves are dated 24<sup>th</sup> c. BC, and currently these are some of the earliest dates for the Elunino sites in the Ob-Irtysh interfluvium; they are synchronous to the dates from the cemetery of Shiderty 10 and the settlement of Shiderty 3 of the north-east Sary-Arka (Merz 2008) and generally correspond with those from the Early Bronze Age kurgans of Aina-Bulak 1 cemetery in the Upper Irtysh River area (Kovalev 2009). The latter suggests that the formation and spread of the Elunino population occurred from West to East. The other three graves analysed were built in the middle of the 22<sup>nd</sup> – early 21<sup>st</sup> c. BC, and they appear to belong to the beginning of the second phase of the Elunino culture, i.e. synchronous to the classical Elunino sites of Berezovaya Luka and Elunino 1 (Grushin 2013).

The dates obtained from the settlement of Shauke 8b (turn of the 3000 to 2000 cal BC) attribute the site to the end of the second stage of Elunino culture, also making it synchronous to the sites of Berezovaya Luka and Elunino 1 (Grushin 2013).

In general, there is a ca. 450 years hiatus between the <sup>14</sup>C dates of the Eneolithic complex of Borly 4 and Early Bronze Elunino site of Shauke 1 which at the moment is likely to be explained by the small number of excavated sites, as well as the lack of the <sup>14</sup>C research in the area. However, the ongoing archaeological work in the region (including the investigation of various buildings of the Borly 4 settlement) might result in discovery of the sites radiocarbon dating to the present chronological hiatus (i.e. 29<sup>th</sup>-25<sup>th</sup> c. BC) and synchronous to e.g. the Early Bronze Age site of New Shulba IX of the Upper Irtysh River region (first half of the 3<sup>rd</sup> mil. BC; Stöllner T. und an. 2013). Further radiocarbon research into the recently discovered sites of the Yamnaya Culture type in the area (Mertz, Mertz 2010) might also fill the present chronological hiatus.

## Conclusions

The study makes significant contributions to several major areas across both environmental science and archaeology of NE Kazakhstan. It represents the first data on freshwater reservoir offsets for the area of entire Kazakhstan, first radiocarbon chronology of the Middle Bronze Age sites of NE Kazakhstan and first palaeoisotopic investigation of the area. The main observations of the study include:

1. The diet of the humans and fauna analysed was based on C<sub>3</sub> foodchains with no evidence of C<sub>4</sub> plants (such as millet) contribution; no major dietary variations have been observed.
2. Aquatic resources apparently were a continuous dietary feature of the Eneolithic to Middle Bronze Age populations of NE Kazakhstan.
3. The first <sup>14</sup>C dates obtained for the Upper and Middle Irtysh River region attribute the Eneolithic period of the area to the 34<sup>th</sup>-30<sup>th</sup> c. BC, and Early Bronze Age – to the 25<sup>th</sup>-20<sup>th</sup> c. BC. As such, the Eneolithic settlement of Borly 4 appears to be synchronous to the sites of Botai and Tersek Culture and to some Eneolithic sites of the south of Western Siberia.
4. At present, there is a ca. 450 years hiatus between the <sup>14</sup>C dates of the Eneolithic complex of Borly 4 and Early Bronze Elunino site of Shauke 1.
5. The maximum fish-herbivore FRO observed in our study equals 301±47 <sup>14</sup>C yrs.

6.  $^{14}\text{C}$  dates from aquatic and human samples from the area apparently need to be interpreted with caution as likely to be affected by the FRO (i.e. appear older).
7. NaOH treatment of bones showed minor but significant effect (0.02 increase in the ratio) on C:N<sub>atomic</sub> ratios of the samples. No significant effect of the treatment on  $\delta^{13}\text{C}$ ,  $\delta^{15}\text{N}$  and collagen yields of the samples has been found.

As a major implication for future work in the area, a larger number of human-herbivore paired samples are needed to be analysed (both for  $^{14}\text{C}$  and  $\delta^{15}\text{N}$ ) to develop a regression model (equation) to predict FRE corrections for the human bone dates, as well as to explore in detail the contribution of fish into the human diet.

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